STATISTICAL DATA ANALYSIS: IN THE CONTEXT OF PROCESS QUALIFICATION AND CONTROL

Elizabeth J. Kelly
Statistical Sciences Group
Los Alamos National Laboratory
MIS Working Group Meeting, October 23

BACKGROUND

DOE-STD-3013-2000 (DOE, 2000) Section 6.1.2.2.2

"Once the process has been qualified, material testing, … , is required … to the extent necessary to show continued process control."

PROCESS QUALIFICATION- Moisture Requirement

- A component of process qualification is the assertion that the calcination process results in items with **moisture content less than 0.5wt%.**
- This assertion must be made in the face of **uncertainty**
- Statistical approach assumes that there are moisture measurement **data** to support this assertion and **quantifies the uncertainty** based on these data.
- Decision makers decide if uncertainty is **acceptable**

UNCERTAINTY QUANTIFICATION

- Statistical analysis quantifies confidence that the average moisture content of canisters resulting from the calcining process is less than a specified level, (0.5wt%).
  - In a perfect world this uncertainty depends only on between item variability, in reality it also depends on possible biases introduced by sampling and measurements
- Statistical analysis can evaluate more than one confidence limit to support assertion
  - For example, 95% confidence that the average is < 0.5wt% and 99% confidence that the average is < 0.6wt% (data not too variable) better than 95% confidence that the average is <0.5wt% and 99% confidence that the average is <1.0wt% (larger tails)

STATISTICAL ANALYSIS FOR PROCESS CONTROL

- Based on a single confirmatory measurement, does the process remain in control, e.g., is the confirmatory sample within the statistical process control limits?
  - These limits depend on sampling and instrument variability, as well as biases and between item variability.
- How many confirmatory samples are needed to have high confidence that the process remains in control?

STATISTICAL APPROACH (documented in STATISTICAL ANALYSES OF MOISTURE MEASUREMENT DATA – A WHITE PAPER LA-UR-02-2388)

Based on mathematical model for moisture measurements

$$X_{ijk} = \mu + P_i + S_{ij} + \varepsilon_{ijk}$$

X_{ijk}	Moisture measurement
μ	Expected moisture after calcining
P_i	Process random variable (rv) with variance σ_P^2
S_{ij}	Sampling rv with bias, β_S and variance σ_S^2
ε_{ijk}	Analytical rv with bias β_ε and variance σ_ε^2

PROCESS QUALIFICATION – EXAMPLE CALCULATIONS

- What is the confidence level for the moisture requirement? (solve for Z)

$$\mu + Z(\%)\sigma_P < 0.5wt\%$$

- Is the 0.5wt% requirement met at the 99% confidence level ?

$$\mu + 2.33\sigma_P < 0.5wt\%$$

PROCESS CONTROL – SAMPLE CALCULATIONS

- Given a single measurement on an item at what level of confidence does the process remain in control? (solve for Z)

$$X_i < \mu + \beta_S + \beta_\varepsilon + Z(\%)\sqrt{\sigma_P^2 + \sigma_S^2 + \sigma_\varepsilon^2}$$

- Given a single measurement on an item does the process remain in control at the 99% level of confidence

$$X_i < \mu + \beta_S + \beta_\varepsilon + 2.33\sqrt{\sigma_P^2 + \sigma_S^2 + \sigma_\varepsilon^2}$$

WHAT MAKES THESE CALCULATIONS DIFFICULT?

- Do not know expected moisture content, variances and biases. Must estimate from data:

$$\mu \quad \sigma_P^2 \quad \sigma_S^2 \quad \sigma_\varepsilon^2 \quad \beta_S \quad \beta_\varepsilon$$

- Statistical estimators are complex (especially if unequal sample sizes), but doable (white paper)
- How good are the data for these estimations?

EXAMPLES – Estimating unknowns μ σ_P^2 σ_S^2 σ_ε^2 β_S β_ε

- Applied successfully to Hanford LOI data for pure materials (one caveat) (white paper)
  - 302 items, 334 measurements
  - Calcining process in control with 99% 99% confidence that average value for all items in this population < 0.23wt.%
  - Upper 99% control limit = 0.24 wt% (process variability dominates). BLO items failed process control check
  - Caveat – possible measurement bias (27 GOI items)
- Applied to limited IGA data, illustrative only (white paper)

WHAT ARE REMAINING STATISTICAL ANALYSIS ISSUES?

- Representativeness ,e.g., Heterogeneity of material, e.g., Sampling uncertainties
- Measurement Biases? No standards for MIS materials. Differences between methods.

WHAT ARE WE DOING TO ADDRESS THESE ISSUES?

- Heterogeneity Issue
  - "Representativeness" summit meeting (October 10)
  - Fractionation Study (Bender, Trujillo)
    - Sieving, particle size fractions
    - Moisture measurements and XRF evaluations made on different fractions
  - Large Scale Surrogate Calcination Study (Dworzak, Gillispie)
    - Looking at impact of chunkiness on moisture uptake
- Bias – ongoing TGA, SFE, IGA studies